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## (54) PRODUCTION OF AQUEOUS (METH)ACRYLAMIDE POLYMER DISPERSION

(57)Abstract:

PURPOSE: To produce an aqueous (meth)acrylamide polymer dispersion that is lowly viscous and excellent in dispersibility and flowability by (co) polymerizing (meth)acrylamide in water containing a specified substance.

CONSTITUTION: (Meth)acrylamide or a mixture thereof with at least one monomer copolymerizable therewith, desirably a water-soluble (meth)allyl compound [e.g. (meth)allyl alcohol] is polymerized in water containing a polyelectrolyte, desirably an alkali metal salt of an acrylic acid polymer (e.g. polysodium acrylate). It is desirable that the ratio of the polyelectrolyte to the polymerizable monomer is 10/90 to 90/10 by weight, and the concentration of the polymerizable monomer in the aqueous solution is 5-50wt.%.

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CLAIMS

[Claim(s)]

[Claim 1] The manufacture approach of the acrylamide system polymer water dispersion characterized for acrylamide by independent or (meta) carrying out the polymerization of the mixture with acrylamide, acrylamide (meta), a kind to copolymerize, or two sorts or more of polymerization nature monomers as a polymerization nature monomer (meta) by underwater [ in which a polyelectrolyte exists ] (meta).

[Claim 2] (Meta) The manufacture approach according to claim 1 characterized by using a water-soluble allyl compound (meta) as acrylamide and a polymerization nature monomer to copolymerize.

[Claim 3] The manufacture approach of claim 2 \*\*\*\*\* which is one sort even if few either that a water-soluble allyl compound (meta) is chosen from allyl alcohol (meta), an allyl compound (meta) sulfonic acid, an allyl compound (meta) carboxylic acid, allylamines (meta), and these salts.

[Claim 4] The manufacture approach according to claim 1 that a polyelectrolyte is the alkali-metal salt of an acrylic-acid system polymer.

[Claim 5] The manufacture approach according to claim 1 that the weight ratios of a polyelectrolyte and a polymerization nature monomer are the former / latter = 10 / 90 - 90/10, and the water-solution concentration of a polymerization nature monomer is 5 - 50 % of the weight.

[Claim 6] The manufacture approach of the acrylamide system polymer characterized by separating with water the acrylamide (meta) system polymer obtained by either of claims 1-5 (meta).

[Claim 7] The manufacture approach of the acrylamide system polymer characterized by drying water and the separated acrylamide (meta) system polymer by claim 7 (meta).

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## DETAILED DESCRIPTION

### [Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the new manufacture approach of an acrylamide (meta) system polymer. Furthermore, it is related with the new manufacture approach of the acrylamide system polymer currently widely used for a thickener, a flocculant, the paper reinforcing agent in pulp and paper industry, the yield improver, the filtration improvement agent, the distributed stabilizer of various suspension solutions, the soil conditioner, etc. in detail (meta).

[0002]

[Description of the Prior Art] The acrylamide system polymer currently used for the above-mentioned purpose from before (meta) is manufactured mainly by the polymerization in the inside of a water solution. In this aqueous polymerization, it is so industrially [ that monomer concentration is high ] advantageous. However, in aqueous polymerization, a product serves as gel which carried out water, and handling becomes it is remarkable and difficult as monomer concentration becomes high. Therefore, although an acrylamide (meta) system polymer is diluted, and it is provided as a low concentration solution or disintegration is dried and carried out, in dealing with it by low concentration, an transportation cost becomes high, and heat energy huge for desiccation disintegration is needed uneconomically, and also it has problems, like insolubilization by thermal three-dimensions bridge formation takes place.

[0003] Moreover, the manufacture approach of the acrylamide system polymer by the emulsion polymerization of a water-in-oil type or the suspension polymerization (meta) in the inside of a hydrophobic solvent is also learned (for example, JP,5-194613,A etc.). However, since the hydrophobic organic solvent used by the emulsion polymerization method or the suspension-polymerization method has a strong odor while being the dangerous substance, it has problems, like a large amount of costs are needed for each manufacturing facility of a polymerization and separation from the problem of work environment.

[0004] Furthermore, it is the manufacture approach by which the precipitate polymerization in a salt water solution also attracts attention (for example, JP,50-70489,A). However, by this approach, the generated polymers adhere and it has the essential fault which finally becomes a big lump. Moreover, the method of performing a precipitate polymerization is also learned in the salt water solution which carries out little content of the polyelectrolyte as protective colloid (JP,62-20511,A). It was difficult for a certain extent to be able to prevent adhesion of the polymers which generated this approach, and to perform a polymerization by high polymerization nature monomer concentration according to a salting-out operation of a thing.

[0005] It is known from before that water soluble polymers, such as an acrylamide (meta) system polymer, will cause other water soluble polymers and interactions underwater, and will produce phase separation on the other hand. For example, if a polyalkylene glycol solution and a polyacrylamide water solution are mixed, phase separation will arise. However, only by applying the interaction reported, the dispersion liquid of the acrylamide system polymer which is rich in dispersibility and a fluidity (meta) are not obtained, and since we were anxious about the effect of the water soluble polymer together put on product performance, they have not resulted in

utilization. Therefore, if the dispersion liquid of the drainage system which is rich in dispersibility and a fluidity by taking the combination of a water soluble polymer into consideration are made, it can be said that it is the gestalt which it should expect an acrylamide (meta) system polymer most.

[0006]

[Means for Solving the Problem] this invention persons repeated examination wholeheartedly about the polymerization of the acrylamide (meta) in a drainage system from such a background paying attention to the interaction of the acrylamide (meta) system polymer and polyelectrolyte in a water solution, the phase separation accompanying it, and an insolubilization phenomenon. Consequently, when the polymerization of the polymerization nature monomer which uses acrylamide (meta) as a principal component was carried out by underwater [ in which a polyelectrolyte exists ], the generated acrylamide (meta) system polymer deposited by the interaction with a polyelectrolyte, and it found out that the aquosity dispersion liquid which are rich in dispersibility and a fluidity by hypoviscosity were obtained.

[0007] While the distributed stability of the water dispersion obtained as polymer concentration becomes high falls and generation of the aggregate under polymerization and adhesion in a container wall become remarkable, a particle will stop however, separation and re-distributing unitedly during standing preservation also by this manufacture approach. Then, this invention persons take notice of the structural effect of the acrylamide (meta) system polymer to the distributed stability of the dispersion liquid formed of the interaction of the acrylamide (meta) system polymer and polyelectrolyte in a water solution. By using the water-soluble allyl compound (meta) which acts as a chain transfer agent as a polymerization nature monomer, in case the polymerization of the mixture of the polymerization nature monomer which uses acrylamide (meta) as a principal component is carried out, as a result of repeating examination further wholeheartedly It found out that the dispersion liquid which the distributed stability of dispersion liquid improves and control the condensation under polymerization and by which redispersible is not spoiled with time by high polymerization nature monomer concentration could be obtained.

[0008] That is, this invention is underwater [ in which a polyelectrolyte exists ], and is independent or (meta) a thing about the manufacture approach of the acrylamide system polymer water dispersion characterized by carrying out the polymerization of the mixture with acrylamide, acrylamide (meta), a kind to copolymerize, or two sorts or more of polymerization nature monomers (meta) about acrylamide as a polymerization nature monomer (meta).

[0009] In this invention, acrylamide is used as an indispensable component as a polymerization nature monomer (meta). (Meta) acrylamide -- the thing of acrylamide and/or methacrylamide -- saying -- this invention -- setting (meta) -- it is the same semantics as the following. Generally, acrylamide is used. \*\* (meta) acrylamide can be used independently, and also it can also be used together with acrylamide (meta), a kind to copolymerize, or two sorts or more of polymerization nature monomers. (Meta) As acrylamide and a polymerization nature monomer to copolymerize, at least one sort chosen from an anionic partial saturation monomer, a cationic partial saturation monomer, a nonionic partial saturation monomer, and a cross-linking partial saturation monomer is raised.

[0010] As an anionic partial saturation monomer, alkali-metal salts, such as sodium salt of organic sulfonic-acids [ , such as dicarboxylic acid; vinyl sulfonic acids such as monocarboxylic acid; maleic acids, such as an acrylic acid, a crotonic acid, and an allyl compound (meta) carboxylic acid, boletic acid, an itaconic acid, and muconic acid, a styrene sulfonic acid, 2-acrylamido-2-methyl propane sulfonic acid, and an allyl compound (meta) sulfonic acid, ]; or these various organic acids and potassium salt, etc. are raised, for example (meta), it is independent in these one sort, or two or more sorts can be used.

[0011] As a cationic partial saturation monomer, to a typical thing, dimethylaminoethyl (meta) acrylate, Diethylaminoethyl (meta) acrylate, dimethylaminopropyl (meta) acrylamide, The partial saturation monomers which have the third class amino groups, such as diethylamino propyl (meta) acrylamide and allylamine (meta), or those hydrochloric acids, The salts of inorganic acids, such as a sulfuric acid and an acetic acid, or an organic acid, or this the third class amino-group

content partial saturation monomer and methyl chloride, The partial saturation monomer containing the quarternary ammonium salt obtained by the reaction with the fourth class-ized agents, such as benzyl chloride, a dimethyl sulfate, and epichlorohydrin, etc. is raised, it is independent in these kinds, or two or more sorts can be used.

[0012] As a nonionic partial saturation monomer, the alkyl ester (carbon numbers 1-8 of an alkyl group) of said anionic partial saturation monomer, acrylonitrile, styrene, vinyl acetate, the methyl vinyl ether, allyl alcohol (meta), etc. are raised. These are independent in a kind or can use two or more sorts.

[0013] Moreover, the following are raised as a typical thing of a cross-linking partial saturation monomer. As a 2 functionality partial saturation monomer, for example, ethylene GURIKORUJI (meta) acrylate, Di(meth)acrylate, such as diethylene GURIKORUJI (meta) acrylate and TORIECHIRENGURIKORUJI (meta) acrylate Methylenebis (meta) acrylamide, ethylene screw (meta) acrylamide, Screw (meta) acrylamides, such as hexa methylenebis (meta) acrylamide Divinyl ester, such as an adipic-acid divinyl and a sebacic-acid divinyl Allyl compound (meta) acrylate and epoxy (meta) acrylate (Meta) Urethane (meta) acrylate, N-methylol (meta) acrylamide, JI (meta) allylamine, JI (meta) allyl compound dimethylannmonium, JI (meta) allyl compound phthalate, a JI (meta) allyl compound KUOREN date, a divinylbenzene, etc. are raised. As a 3 functionality monomer, moreover, 1, 3, 5-Tori (meta) acryloyl hexahydro-S-triazine, Tori (meta) allyl compound isocyanurate, the Tori (meta) allylamine, Tori (meta) allyl compound trimellitate, N, and N-JI (meta) allyl compound (meta) acrylamide etc. as a 4 functionality partial saturation monomer Tetramethylolmethane tetrapod (meta) acrylate, tetrapod (meta) allyl compound pyromellitate, N and N, N', N'-tetrapod (meta) allyl compound-1,4-diaminobutane, a tetrapod (meta) allylamine salt, tetrapod (meta) allyloxy ethane, etc. are raised. These are independent in a kind or can use two or more sorts.

[0014] the total mol sum of the polymerization nature monomer in which the amount of the aforementioned (meta) acrylamide and the polymerization nature monomer used to copolymerize contains acrylamide -- usually -- less than [ 50 mol % ] -- it is less than [ 20 mol % ] preferably. (Meta) Since the rate of acrylamide decreases when the amount of acrylamide and the polymerization nature monomer used to copolymerize exceeds 50-mol % (meta), the phase separation by the interaction with a polyelectrolyte stops arising, and the dispersion liquid of an acrylamide (meta) system polymer are no longer obtained. in addition, the application of the acrylamide system polymer with which an anionic partial saturation monomer, a cationic partial saturation monomer, a nonionic partial saturation monomer, and a cross-linking partial saturation monomer can be used together with acrylamide (meta) combining a kind or two sorts or more, respectively, and the operating rate is obtained (meta) etc. -- taking into consideration -- the total mol sum of a polymerization nature monomer -- usually -- 50-mol% of within the limits -- proper -- determining -- \*\*\*\*ing . However, when using a cross-linking partial saturation monomer, it is good less than [ of the total mol sum of a polymerization nature monomer / usual 10 mol % ], and to consider as 0.01-5-mol % preferably. Dispersion liquid are not obtained in order that the whole polymerization solution may construct a bridge, in exceeding ten-mol %.

[0015] Moreover, by this invention, by using a water-soluble allyl compound (meta) as a polymerization nature monomer copolymerized with acrylamide (meta), even when polymerization nature monomer concentration is high, the distributed stability of dispersion liquid can improve, the condensation under polymerization can be controlled, and the dispersion liquid by which redispersible is not spoiled with time can be obtained. It is the effectiveness looked at by only the water-soluble allyl compound (meta) as a polymerization nature monomer about a detail although it is not clear. The branching structure and end group structure which are formed during a polymerization by using the water-soluble allyl compound (meta) which has a chain transfer operation reduce remarkably the viscosity of the water solution of an acrylamide (meta) system macromolecule. While controlling fusion between the particles in the early stages of a polymerization, and condensation, it is thought that these structural factors are also essentially raising the dispersibility of the insoluble ghost produced by the interaction with a polyelectrolyte. In addition, distributed stability etc. cannot be raised even if it uses the isopropyl alcohol generally used as a chain transfer agent.

[0016] As this water-soluble allyl compound (meta), various kinds of things of an anionic partial saturation monomer, a cationic partial saturation monomer, a nonionic partial saturation monomer, or a cross-linking partial saturation monomer are raised. Allyl alcohol (meta), an allyl compound (meta) carboxylic acid, an allyl compound (meta) sulfonic acid, allylamine (meta), or its salts is raised, and these are independent in a kind or, specifically, can use two or more sorts. Also in these, it is desirable in an allyl compound (meta) sulfonic acid or its salts, and especially a meta-allyl compound sulfonate is effective. Since the molecular weight of the acrylamide system polymer obtained by these use (meta) is changed sharply, the amount of the water-soluble allyl compound used is determined in consideration of the application. usually, the rate of a water-soluble allyl compound -- the total mol sum of a polymerization nature monomer -- receiving -- less than [ 10 mol % ] -- desirable -- more than 0.1 mol % -- less than [ 10 mol % ] -- it is less than [ 5 mol % ] more than in 0.5 mol % more preferably. When there is nothing and it exceeds ten-mol %, a molecular weight fall becomes almost practical [ the effectiveness to improvement in distributed stability / become remarkable and ] less less than [ 0.1 mol % ].

[0017] In this invention, into a giant molecule, the polyelectrolyte which makes it exist underwater at the time of manufacture of an acrylamide (meta) system polymer has carboxylate and/or a sulfonate, and means what produces phase separation by the interaction with the acrylamide (meta) system polymer to generate. As such a polyelectrolyte, it is independent in a kind of the anionic partial saturation monomer of said instantiation, or the salt of the polymer obtained by carrying out the polymerization of the two or more sorts is raised, for example. Especially as a polyelectrolyte of this invention, the salt of the acrylic-acid polymer which used the acrylic acid as an anionic partial saturation monomer is desirable.

[0018] Moreover, although especially the class of salt of a polymer is not limited, in this invention, alkali-metal salts, such as sodium salt or potassium salt, are desirable. Therefore, especially as a polyelectrolyte of this invention, the alkali-metal salt of an acrylic-acid polymer is desirable. the neutralization index of the carboxylic acid of a polyelectrolyte, and/or a sulfonic acid -- usually -- 50-100-mol % -- it is 70-100-mol % preferably. Formation of this carboxylate and/or a sulfonate may be performed in the phase of an anionic partial saturation monomer, and you may carry out, after manufacturing a polymer.

[0019] Moreover, if the polyelectrolyte of this invention is within the limits which produces phase separation by the interaction with an acrylamide (meta) system polymer, it will be used together with an anionic partial saturation monomer, copolymerizes at least one sort chosen from amide group content partial saturation monomers, such as a cationic partial saturation monomer of said instantiation, a nonionic partial saturation monomer, and acrylamide (meta), and may be obtained. usually, the total mol sum of the partial saturation monomer from which the rate of the copolymerization monomer used together with an anionic partial saturation monomer constitutes a polyelectrolyte -- receiving -- usually -- less than [ 30 mol % ] -- it is less than [ 10 mol % ] preferably. In exceeding 30-mol %, it is hard to produce phase separation by the interaction with an acrylamide (meta) system polymer.

[0020] Although it cannot generally \*\*\*\*\* since the weight average molecular weight of the polyelectrolyte used for this invention will not be restricted especially if it insolubilizes in water by mixing with an acrylamide (meta) system polymer, but it changes with the presentation of an acrylamide (meta) system polymer, conditions of a polymerization reaction, etc., weight average molecular weight is 100,000 or less, and 5000-50000 are usually desirable. When lower than 5000, phase separation cannot happen easily, and the dispersion liquid of an acrylamide (meta) system polymer are not obtained. When exceeding 100,000, the viscosity of a system becomes moreover, less practical [ increase and ].

[0021] Next, the manufacture approach of the acrylamide (meta) system polymer of this invention is explained in detail. although the amount of the polyelectrolyte used changes with the presentation of a polyelectrolyte, molecular weight, the presentation ratio of a polymerization nature monomer and polymerization concentration, and other polymerization conditions -- usually -- the weight ratio of a polyelectrolyte and a polymerization nature monomer -- usually -- the former / latter = 10 / 90 - 90/10 -- it is 20 / 80 - 50/50 preferably. If the rate of a polyelectrolyte separates from this range, separation of the acrylamide system polymer obtained

by carrying out a polymerization (meta) will not take place, and dispersion liquid will not be obtained.

[0022] Moreover, the water-solution concentration of a polymerization nature monomer is usually 15 – 40 % of the weight preferably five to 50% of the weight. The dispersibility of the dispersion liquid which will be obtained if water-solution concentration exceeds 50 % of the weight, and a fluidity become less practical [ fall and ]. Moreover, at less than 5 % of the weight, the phase separation by the interaction of a polyelectrolyte and an acrylamide (meta) system polymer stops arising, and dispersion liquid are not obtained.

[0023] Except for making a polyelectrolyte exist, various well-known approaches and the same approach can be underwater used for the method of carrying out the polymerization of said polymerization nature monomer, and obtaining an acrylamide (meta) system polymer conventionally. For example, a polymerization nature monomer, a polyelectrolyte, and water are taught to a predetermined reaction container, the usual radical polymerization initiators, such as a redox system polymerization initiator of the form which combined persulfate, such as potassium persulfate and ammonium persulfate, or these and the reducing agent like a sodium hydrogensulfite, can be added, and the dispersion liquid of the acrylamide (meta) system polymer of this invention can be obtained by the approach of warming under churning.

[0024] The amount of the radical polymerization initiator used is set up in consideration of the amount of the water-soluble allyl compound (meta) used etc. in the range which can respond to the molecular weight of the acrylamide system polymer obtained (meta). Usually, the amount of the radical polymerization initiator used is usually 0.01–10–mol % to a polymerization nature monomer. moreover, pH of the polyelectrolyte water solution containing the polymerization nature monomer at the time of a polymerization — usually — 6–10 — it is 7–8 preferably, and it is necessary to set up the neutralization index of the carboxylic acid of said polyelectrolyte, and/or a sulfonic acid so that it may go into this range Since it produces hydrolysis of an acrylamide system polymer in phase separation's stopping being able to happen easily when pH is lower than 6, and exceeding 10 (meta), it is not desirable. Although the temperature of a polymerization changes with presentations and those concentration of the class of radical polymerization initiator, a polymerization nature monomer, and a polyelectrolyte and it cannot generally \*\*\*\*\*, it is usually within the limits of 25–90 degrees C, and a polymerization is performed to the bottom of such a condition for 1 to 12 hours. In addition, on the occasion of a polymerization, chain transfer agents other than a water-soluble allyl compound (meta), a surfactant, a salt, etc. can be used for arbitration as an additive if needed. Although the amount of the additive used changes with applications of the acrylamide system polymer obtained (meta) etc. and cannot be limited especially, generally it is 10 or less % of the weight to a polymerization nature monomer.

[0025] The weight average molecular weight of the acrylamide (meta) system polymer obtained in this way is usually 100,000 to about 20 million. Moreover, an acrylamide (meta) system polymer produces phase separation by the interaction with a polyelectrolyte, and is obtained as dispersion liquid of both mixed constituent particle. The viscosity of the dispersion liquid of the acrylamide system polymer containing this polyelectrolyte (meta) is usually about (25 degrees C) 100–10000cps in 20% of concentration. The distributed object of the acrylamide system polymer containing such a polyelectrolyte (meta) is a gel object which usually contains 30 – 70% of the weight of water. These have dissociated with water and are rich in dispersibility and a fluidity. Although the weight ratio of the polyelectrolyte in a gel object and an acrylamide (meta) system polymer changes with conditions of the preparation ratio of each component, or a polymerization, they are usually the former / latter =50 / 50 – 10/90. In addition, although surfacing of a distributed object and separation may produce the obtained distributed object if it carries out long duration gentle placement, as for the distributed object using a water-soluble allyl compound (meta), it is possible as a polymerization nature monomer to make churning re-distribute easily.

[0026] Moreover, the water gel particle of the acrylamide system macromolecule containing a polyelectrolyte (meta) is separable from dispersion liquid with water easily by centrifugal separation, filtration, or other suitable separation approaches. Moreover, water and the separated

water gel particle have little moisture content, it is more nearly granular than the water gel polymerization object obtained by the usual aqueous polymerization, and since surface area also becomes large, it is easy to dry. Generally, warm air (for example, 30–90 degrees C) can perform desiccation with the fluid bed etc.

[0027]

[Function] The aquosity dispersion liquid of the high-concentration acrylamide (meta) system polymer which is rich in dispersibility and a fluidity with this invention can be obtained. Although the detail of the operation cannot fully be solved, since the insolubilization phenomenon by the interaction of the acrylamide (meta) system polymer and polyelectrolyte in a water solution is applied to formation of dispersion liquid, it is hard to be influenced of a presentation by the polymer monomer, and distributed stability is good and it is the description that the aquosity dispersion liquid of the hypoviscosity which is rich in a fluidity can be obtained. Moreover, an acrylamide (meta) system polymer is easily separable as a water particle containing a polyelectrolyte. A water particle is not only easy handling, but the solubility over water is good and it can raise as a description that dilution time amount can be shortened. In addition, when a cross-linking partial saturation monomer is used as a polymerization nature monomer, the bridge formation particle dispersion liquid of an acrylamide (meta) system polymer are obtained.

Filtration can separate this bridge formation particle easily, and a polyelectrolyte can be removed by washing with water. Furthermore, in manufacture of the dispersion liquid by the interaction of an acrylamide (meta) system polymer and a polyelectrolyte, also when the polymerization nature monomer of the distributed object using a water-soluble allyl compound (meta) is high concentration as a polymerization nature monomer, the condensation between the particles under polymerization can be controlled, and large improvement in distributed stability can be realized.

[0028]

[Example] Although an example explains this invention concretely below, this invention is not limited to this. In addition, % in each example is weight criteria, and the molecular weight of an acrylamide (meta) system polymer is the value which measured using drainage system gel PAMINEESHON chroma log RAFI, and was converted into polyethylene-glycol molecular weight.

[0029] Example 1 acrylamide 100g, 576.9g (pH8.0, weight average molecular weight 19000

[ about ]) of 26-% of the weight sodium polyacrylate water solutions, and 92.3g (12.7 % of the weight of concentration of a polymerization nature monomer) of ion exchange water A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, 10g of ammonium persulfate water solutions and 10g of 1-% of the weight sodium-hydrogensulfite water solutions are added 1% of the weight through nitrogen gas, and a polymerization is carried out at 40 degrees C under churning for 10 hours. The polymer distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 5500cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 8500000.

[0030] Example 2 acrylamide 170g, 7.2g of acrylic acids, 3.2g of sodium hydroxides, 395.3g (pH8.3, weight average molecular weight 15000 [ about ]) of sodium polyacrylate water solutions, and 274.3g (20.6 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, isopropyl alcohol 7.5g and 14.2g of 10-% of the weight ammonium persulfate water solutions, and 5.7g of 10-% of the weight sodium-sulfite water solutions are added through nitrogen gas, and a polymerization is carried out at 40 degrees C under churning for 4 hours. The polymer underwater distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 1200cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 5400000.

[0031] Example 3 acrylamide 100g, 37.5g of the fourth class ghost water solutions of 81-% of the weight dimethylaminoethyl methacrylate methyl chloride, 232.6g (pH8.3, weight average molecular weight 15000 [ about ]) of sodium polyacrylate water solutions, and 281.3g (19.9 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A



stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, isopropyl alcohol 4.7g and 0.38g of 10-% of the weight ammonium persulfate water solutions, and 0.14g of 10-% of the weight sodium-sulfite water solutions are added through nitrogen gas, and a polymerization is carried out at 80 degrees C under churning for 4 hours. The polymer distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 200cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 12 million.

[0032] Example 4 acrylamide 200g, 8.5g of acrylic acids, 3.8g of sodium hydroxides, 199.3g (pH8.3, weight average molecular weight 15000 [ about ]) of sodium polyacrylate water solutions, and 231.6g (30.5 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, isopropyl alcohol 9g and 16.7g of 10-% of the weight ammonium persulfate water solutions, and 6.8g of 10-% of the weight sodium-sulfite water solutions are added through nitrogen gas, and a polymerization is carried out at 80 degrees C under churning for 4 hours. The polymer distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 6800cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 2100000.

[0033] Example 5 acrylamide 100g, isopropyl alcohol 3.3g (3.9mol for acrylamide %), 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 164.1g (19.6 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer was obtained by adding 8.0g of ammonium persulfate water solutions, and 3.3g of 10-% of the weight sodium-pyrosulfite water solutions 10% of the weight except for the dissolved oxygen in liquid through nitrogen gas, and carrying out a polymerization at 45 degrees C under churning for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 3800cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 2 million.

[0034] The particle which consists of an acrylamide system polymer and sodium polyacrylate was separated from dispersion liquid by carrying out filtration under reduced pressure (100 or more mmHg) of the acrylamide system polymer dispersion liquid compounded in the example 6 example 2 using a glass filter. The particle was the water gel which contained 43% of the weight of water with the mixture of an acrylamide system polymer (85 % of the weight) and sodium polyacrylate (15 % of the weight). The obtained granular water gel could be ground easily and has been dried in about 30 minutes 65-degree-C time amount with the fluid bed dryer.

[0035] While putting 100g of sodium acetate, and 200g of ion exchange water into the reaction container used in the example of comparison 1 example 1 as acrylamide 100g and a low-molecular electrolyte and keeping at 35 degrees C When 10g of ammonium persulfate water solutions and 10g of 1-% of the weight sodium-hydrogensulfite water solutions were added 1% of the weight except for dissolved oxygen through nitrogen gas and the polymerization was performed at 50 degrees C under churning, the viscosity of reaction mixture became extremely high, and even if it carried out the polymerization for 10 hours, dispersion liquid were not obtained with the gel lump.

[0036] In the reaction container used in the example of comparison 2 example 1, acrylamide 75g, 42.5g of ammonium sulfates, 42.5g of diammonium hydrogenphosphate, 7.5g (weight average molecular weight 15000 [ about ]) of sodium polyacrylate, and 200g of ion exchange water are put in. If 0.75g of ammonium persulfate water solutions and 0.7g of 1-% of the weight sodium-hydrogensulfite water solutions are added 1% of the weight except for dissolved oxygen through nitrogen gas and a polymerization is performed at 40 degrees C under churning, keeping at 35 degrees C, the viscosity of reaction mixture is with a gel lump, even if it becomes extremely high and carries out a polymerization for 10 hours. Dispersion liquid were not obtained.

[0037] Example 7 acrylamide 100g, allyl alcohol 3.2g (3.9mol for acrylamide %), 232.6g (pH8.0,

weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 164.3g (20.2 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer distributed by the shape of a particle was obtained by adding 3.3g of pyrosulfurous acid hydrogen sodium water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 680cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 1090000.

[0038] Example 8 acrylamide 100g, 7.9g (3.9mol for acrylamide %) of sodium allylsulfonate, 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 159.5g (21.1 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer underwater distributed by the shape of a particle was obtained by adding 3.3g of sodium-pyrosulfite water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 1450cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 1740000.

[0039] Example 9 acrylamide 100g, 1.1g (0.5mol for acrylamide %) of meta-sodium allylsulfonate, 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 166.3g (19.8 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer distributed by the shape of a particle was obtained by adding 3.3g of sodium-pyrosulfite water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 460cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 1190000.

[0040] Example 10 acrylamide 100g, 8.6g (3.9mol for acrylamide %) of meta-sodium allylsulfonate, 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 58.9g (26.4 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer distributed by the shape of a particle was obtained by adding 3.3g of sodium-pyrosulfite water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 5900cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 120000.

[0041] Example 11 acrylamide 100g, methylenebis acrylamide 1.1g (0.5mol for acrylamide %), 8.6g (3.9mol for acrylamide %) of meta-sodium allylsulfonate, 464.5g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 424.7g (10.9 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The water dispersion of an acrylamide cross-linked-polymer particle was obtained by adding 8.0g of ammonium persulfate water solutions, and 3.3g of 10-% of the weight sodium-sulfite water solutions 10% of the weight except for the dissolved oxygen in liquid through nitrogen gas, and carrying out a polymerization at 45 degrees C under churning for 3 hours, keeping at 20 degrees C. Furthermore, these dispersion liquid were carried out the \*\* exception, and the true spherical particle of a bridge formation acrylamide system

polymer with a particle diameter of about 100 microns was obtained by drying among washing and the type dryer of the \*\* style with a lot of water in 105 degrees C and 2 hours.

[0042] The situation under polymerization in an example 5 and examples 7-11, the situation after the standing of the dispersion liquid of the obtained acrylamide system polymer, the viscosity after re-distribution, and redispersible evaluation are shown in Table 1.

[0043] : (Situation under polymerization) At the time of polymerization termination, condensation of the distributed object in the inside of dispersion liquid, and a \*\*\*\* wing and a reaction container wall surface and adhesion were observed by viewing, and were evaluated.

[0044] : (Situation after standing) The obtained dispersion liquid were put at 25 degrees C after polymerization termination for 48 hours, and condensation of the distributed object in the inside of dispersion liquid and a container wall surface and adhesion were observed by viewing, and were evaluated.

[0045] : (Viscosity after re-distribution) After putting dispersion liquid at 25 degrees C for 48 hours, the whole was agitated with the stirrer for 5 minutes, and the viscosity of dispersion liquid was measured by the Brookfield viscometer. In addition, all over Table 1, the result of having measured the viscosity of the obtained dispersion liquid by the Brookfield viscometer after polymerization termination is shown as (viscosity in front of standing).

[0046] : (Redispersible evaluation) Viewing observed and estimated the situation after re-distribution.

[0047]

[Table 1]

|       | 重合中の状況 | 静置後の状況 | 静置前の<br>粘度<br>(c p) | 再分散後<br>の粘度<br>(c p) | 再分散性<br>の評価 |
|-------|--------|--------|---------------------|----------------------|-------------|
| 実施例 5 | 凝集物付着  | 分離、凝集  | 3 8 0 0             | 2 0 5 0              | 不良          |
| 実施例 7 | 付着物なし  | 分離     | 6 2 0               | 5 5 0                | 良好          |
| 実施例 8 | 付着物なし  | 分離     | 1 4 5 0             | 1 4 0 0              | 良好          |
| 実施例 9 | 付着物なし  | 分離     | 4 6 0               | 4 7 5                | 良好          |
| 実施例10 | 付着物なし  | 変化なし   | 5 9 0 0             | 6 0 0 0              | 良好          |
| 実施例11 | 付着物なし  | 分離     | 1 5 8               | 1 5 0                | 良好          |

[0048]

[Effect of the Invention] This invention is the new manufacture approach of the acrylamide system polymer in the inside of the water solution containing a polyelectrolyte, and can obtain the aquosity dispersion liquid which are rich in dispersibility and a fluidity by hypoviscosity. Moreover, also when a polymerization nature monomer is high concentration, formation of the aggregate under polymerization is controlled, and since the aquosity dispersion liquid of the hypoviscosity which kept dispersibility with time can be obtained, there is the following effectiveness.

[0049] (1) Since it can manufacture easily without an inflammable organic solvent, it is cheap, and also economically, excel. (2) Since it is dispersion liquid of a fluid high acrylamide system polymer and is high concentration in hypoviscosity, an transportation cost becomes cheap and is economically excellent. (3) A granular acrylamide system polymer can be separated easily and the water particle which is excellent in the solubility to water can be offered.

[0050] Therefore, the aquosity dispersion liquid of the acrylamide system polymer manufactured by this invention can be widely used for a thickener, a flocculant, the paper reinforcing agent in pulp and paper industry, a yield improver, a filtration improvement agent, the distributed stabilizer of various suspension solutions, a soil conditioner, etc., and are very effective in them. Moreover,

the bridge formation particle of the acrylamide system polymer obtained when a cross-linking partial saturation monomer is used as a polymerization nature monomer (meta) has a hydrophilic property and absorptivity depending on the polymerization nature monomer presentation, and can use them as an absorptivity grant agent, an antistatic agent, etc. to fluid amelioration agents, such as a high absorptivity polymer and a drainage system coating, plastics, etc.

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TECHNICAL FIELD

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[Industrial Application] This invention relates to the new manufacture approach of an acrylamide (meta) system polymer. Furthermore, it is related with the new manufacture approach of the acrylamide system polymer currently widely used for a thickener, a flocculant, the paper reinforcing agent in pulp and paper industry, the yield improver, the filtration improvement agent, the distributed stabilizer of various suspension solutions, the soil conditioner, etc. in detail (meta).

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EFFECT OF THE INVENTION

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[Effect of the Invention] This invention is the new manufacture approach of the acrylamide system polymer in the inside of the water solution containing a polyelectrolyte, and can obtain the aquosity dispersion liquid which are rich in dispersibility and a fluidity by hypoviscosity. Moreover, also when a polymerization nature monomer is high concentration, formation of the aggregate under polymerization is controlled, and since the aquosity dispersion liquid of the hypoviscosity which kept dispersibility with time can be obtained, there is the following effectiveness.

[0049] (1) Since it can manufacture easily without an inflammable organic solvent, it is cheap, and also economically, excel. (2) Since it is dispersion liquid of a fluid high acrylamide system polymer and is high concentration in hypoviscosity, an transportation cost becomes cheap and is economically excellent. (3) A granular acrylamide system polymer can be separated easily and the water particle which is excellent in the solubility to water can be offered.

[0050] Therefore, the aquosity dispersion liquid of the acrylamide system polymer manufactured by this invention can be widely used for a thickener, a flocculant, the paper reinforcing agent in pulp and paper industry, a yield improver, a filtration improvement agent, the distributed stabilizer of various suspension solutions, a soil conditioner, etc., and are very effective in them. Moreover, the bridge formation particle of the acrylamide system polymer obtained when a cross-linking partial saturation monomer is used as a polymerization nature monomer (meta) has a hydrophilic property and absorptivity depending on the polymerization nature monomer presentation, and can use them as an absorptivity grant agent, an antistatic agent, etc. to fluid amelioration agents, such as a high absorptivity polymer and a drainage system coating, plastics, etc.

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## TECHNICAL PROBLEM

[Description of the Prior Art] The acrylamide system polymer currently used for the above-mentioned purpose from before (meta) is manufactured mainly by the polymerization in the inside of a water solution. In this aqueous polymerization, it is so industrially [ that monomer concentration is high ] advantageous. However, in aqueous polymerization, a product serves as gel which carried out water, and handling becomes it is remarkable and difficult as monomer concentration becomes high. Therefore, although an acrylamide (meta) system polymer is diluted, and it is provided as a low concentration solution or disintegration is dried and carried out, in dealing with it by low concentration, an transportation cost becomes high, and heat energy huge for desiccation disintegration is needed uneconomically, and also it has problems, like insolubilization by thermal three-dimensions bridge formation takes place.

[0003] Moreover, the manufacture approach of the acrylamide system polymer by the emulsion polymerization of a water-in-oil type or the suspension polymerization (meta) in the inside of a hydrophobic solvent is also learned (for example, JP,5-194613,A etc.). However, since the hydrophobic organic solvent used by the emulsion polymerization method or the suspension-polymerization method has a strong odor while being the dangerous substance, it has problems, like a large amount of costs are needed for each manufacturing facility of a polymerization and separation from the problem of work environment.

[0004] Furthermore, it is the manufacture approach by which the precipitate polymerization in a salt water solution also attracts attention (for example, JP,50-70489,A). However, by this approach, the generated polymers adhere and it has the essential fault which finally becomes a big lump. Moreover, the method of performing a precipitate polymerization is also learned in the salt water solution which carries out little content of the polyelectrolyte as protective colloid (JP,62-20511,A). It was difficult for a certain extent to be able to prevent adhesion of the polymers which generated this approach, and to perform a polymerization by high polymerization nature monomer concentration according to a salting-out operation of a thing.

[0005] It is known from before that water soluble polymers, such as an acrylamide (meta) system polymer, will cause other water soluble polymers and interactions underwater, and will produce phase separation on the other hand. For example, if a polyalkylene glycol solution and a polyacrylamide water solution are mixed, phase separation will arise. However, only by applying the interaction reported, the dispersion liquid of the acrylamide system polymer which is rich in dispersibility and a fluidity (meta) are not obtained, and since we were anxious about the effect of the water soluble polymer together put on product performance, they have not resulted in utilization. Therefore, if the dispersion liquid of the drainage system which is rich in dispersibility and a fluidity by taking the combination of a water soluble polymer into consideration are made, it can be said that it is the gestalt which it should expect an acrylamide (meta) system polymer most.

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## MEANS

[Means for Solving the Problem] this invention persons repeated examination wholeheartedly about the polymerization of the acrylamide (meta) in a drainage system from such a background paying attention to the interaction of the acrylamide (meta) system polymer and polyelectrolyte in a water solution, the phase separation accompanying it, and an insolubilization phenomenon. Consequently, when the polymerization of the polymerization nature monomer which uses acrylamide (meta) as a principal component was carried out by underwater [ in which a polyelectrolyte exists ], the generated acrylamide (meta) system polymer deposited by the interaction with a polyelectrolyte, and it found out that the aqueous dispersion liquid which are rich in dispersibility and a fluidity by hypoviscosity were obtained.

[0007] While the distributed stability of the water dispersion obtained as polymer concentration becomes high falls and generation of the aggregate under polymerization and adhesion in a container wall become remarkable, a particle will stop however, separation and re-distributing unitedly during standing preservation also by this manufacture approach. Then, this invention persons take notice of the structural effect of the acrylamide (meta) system polymer to the distributed stability of the dispersion liquid formed of the interaction of the acrylamide (meta) system polymer and polyelectrolyte in a water solution. By using the water-soluble allyl compound (meta) which acts as a chain transfer agent as a polymerization nature monomer, in case the polymerization of the mixture of the polymerization nature monomer which uses acrylamide (meta) as a principal component is carried out, as a result of repeating examination further wholeheartedly It found out that the dispersion liquid which the distributed stability of dispersion liquid improves and control the condensation under polymerization and by which redispersible is not spoiled with time by high polymerization nature monomer concentration could be obtained.

[0008] That is, this invention is underwater [ in which a polyelectrolyte exists ], and is independent or (meta) a thing about the manufacture approach of the acrylamide system polymer water dispersion characterized by carrying out the polymerization of the mixture with acrylamide, acrylamide (meta), a kind to copolymerize, or two sorts or more of polymerization nature monomers (meta) about acrylamide as a polymerization nature monomer (meta).

[0009] In this invention, acrylamide is used as an indispensable component as a polymerization nature monomer (meta). (Meta) acrylamide -- the thing of acrylamide and/or methacrylamide -- saying -- this invention -- setting (meta) -- it is the same semantics as the following. Generally, acrylamide is used. \*\* (meta) acrylamide can be used independently, and also it can also be used together with acrylamide (meta), a kind to copolymerize, or two sorts or more of polymerization nature monomers. (Meta) As acrylamide and a polymerization nature monomer to copolymerize, at least one sort chosen from an anionic partial saturation monomer, a cationic partial saturation monomer, a nonionic partial saturation monomer, and a cross-linking partial saturation monomer is raised.

[0010] As an anionic partial saturation monomer, alkali-metal salts, such as sodium salt of organic sulfonic-acids [ , such as dicarboxylic acid; vinyl sulfonic acids such as monocarboxylic acid; maleic acids, such as an acrylic acid, a crotonic acid, and an allyl compound (meta) carboxylic acid, boletic acid, an itaconic acid, and muconic acid, a styrene sulfonic acid, 2-



acrylamido-2-methyl propane sulfonic acid, and an allyl compound (meta) sulfonic acid, ]; or these various organic acids and potassium salt, etc. are raised, for example (meta), it is independent in these one sort, or two or more sorts can be used.

[0011] As a cationic partial saturation monomer, to a typical thing, dimethylaminoethyl (meta) acrylate, Diethylaminoethyl (meta) acrylate, dimethylaminopropyl (meta) acrylamide, The partial saturation monomers which have the third class amino groups, such as diethylamino propyl (meta) acrylamide and allylamine (meta), or those hydrochloric acids, The salts of inorganic acids, such as a sulfuric acid and an acetic acid, or an organic acid, or this the third class amino-group content partial saturation monomer and methyl chloride, The partial saturation monomer containing the quarternary ammonium salt obtained by the reaction with the fourth class-ized agents, such as benzyl chloride, a dimethyl sulfate, and epichlorohydrin, etc. is raised, it is independent in these kinds, or two or more sorts can be used.

[0012] As a nonionic partial saturation monomer, the alkyl ester (carbon numbers 1-8 of an alkyl group) of said anionic partial saturation monomer, acrylonitrile, styrene, vinyl acetate, the methyl vinyl ether, allyl alcohol (meta), etc. are raised. These are independent in a kind or can use two or more sorts.

[0013] Moreover, the following are raised as a typical thing of a cross-linking partial saturation monomer. As a 2 functionality partial saturation monomer, for example, ethylene GURIKORUJI (meta) acrylate, Di(meth)acrylate, such as diethylene GURIKORUJI (meta) acrylate and TORIECHIRENGURIKORUJI (meta) acrylate Methylenebis (meta) acrylamide, ethylene screw (meta) acrylamide, Screw (meta) acrylamides, such as hexa methylenebis (meta) acrylamide Divinyl ester, such as an adipic-acid divinyl and a sebacic-acid divinyl Allyl compound (meta) acrylate and epoxy (meta) acrylate (Meta) Urethane (meta) acrylate, N-methylol (meta) acrylamide, JI (meta) allylamine, JI (meta) allyl compound dimethylannmonium, JI (meta) allyl compound phthalate, a JI (meta) allyl compound KUOREN date, a divinylbenzene, etc. are raised. As a 3 functionality monomer, moreover, 1, 3, 5-Tori (meta) acryloyl hexahydro-S-triazine, Tori (meta) allyl compound isocyanurate, the Tori (meta) allylamine, Tori (meta) allyl compound trimellitate, N, and N-JI (meta) allyl compound (meta) acrylamide etc. as a 4 functionality partial saturation monomer Tetramethylolmethane tetrapod (meta) acrylate, tetrapod (meta) allyl compound pyromellitate, N and N, N', N'-tetrapod (meta) allyl compound-1,4-diaminobutane, a tetrapod (meta) allylamine salt, tetrapod (meta) allyloxy ethane, etc. are raised. These are independent in a kind or can use two or more sorts.

[0014] the total mol sum of the polymerization nature monomer in which the amount of the aforementioned (meta) acrylamide and the polymerization nature monomer used to copolymerize contains acrylamide -- usually -- less than [ 50 mol % ] -- it is less than [ 20 mol % ] preferably. (Meta) Since the rate of acrylamide decreases when the amount of acrylamide and the polymerization nature monomer used to copolymerize exceeds 50-mol % (meta), the phase separation by the interaction with a polyelectrolyte stops arising, and the dispersion liquid of an acrylamide (meta) system polymer are no longer obtained. in addition, the application of the acrylamide system polymer with which an anionic partial saturation monomer, a cationic partial saturation monomer, a nonionic partial saturation monomer, and a cross-linking partial saturation monomer can be used together with acrylamide (meta) combining a kind or two sorts or more, respectively, and the operating rate is obtained (meta) etc. -- taking into consideration -- the total mol sum of a polymerization nature monomer -- usually -- 50-mol% of within the limits -- proper -- determining -- \*\*\*\*ing . However, when using a cross-linking partial saturation monomer, it is good less than [ of the total mol sum of a polymerization nature monomer / usual 10 mol % ], and to consider as 0.01-5-mol % preferably. Dispersion liquid are not obtained in order that the whole polymerization solution may construct a bridge, in exceeding ten-mol %.

[0015] Moreover, by this invention, by using a water-soluble allyl compound (meta) as a polymerization nature monomer copolymerized with acrylamide (meta), even when polymerization nature monomer concentration is high, the distributed stability of dispersion liquid can improve, the condensation under polymerization can be controlled, and the dispersion liquid by which redispersible is not spoiled with time can be obtained. It is the effectiveness looked at by only the water-soluble allyl compound (meta) as a polymerization nature monomer about a detail

although it is not clear. The branching structure and end group structure which are formed during a polymerization by using the water-soluble allyl compound (meta) which has a chain transfer operation reduce remarkably the viscosity of the water solution of an acrylamide (meta) system macromolecule. While controlling fusion between the particles in the early stages of a polymerization, and condensation, it is thought that these structural factors are also essentially raising the dispersibility of the insoluble ghost produced by the interaction with a polyelectrolyte. In addition, distributed stability etc. cannot be raised even if it uses the isopropyl alcohol generally used as a chain transfer agent.

[0016] As this water-soluble allyl compound (meta), various kinds of things of an anionic partial saturation monomer, a cationic partial saturation monomer, a nonionic partial saturation monomer, or a cross-linking partial saturation monomer are raised. Allyl alcohol (meta), an allyl compound (meta) carboxylic acid, an allyl compound (meta) sulfonic acid, allylamine (meta), or its salts is raised, and these are independent in a kind or, specifically, can use two or more sorts. Also in these, it is desirable in an allyl compound (meta) sulfonic acid or its salts, and especially a meta-allyl compound sulfonate is effective. Since the molecular weight of the acrylamide system polymer obtained by these use (meta) is changed sharply, the amount of the water-soluble allyl compound used is determined in consideration of the application. usually, the rate of a water-soluble allyl compound -- the total mol sum of a polymerization nature monomer -- receiving -- less than [ 10 mol % ] -- desirable -- more than 0.1 mol % -- less than [ 10 mol % ] -- it is less than [ 5 mol % ] more than in 0.5 mol % more preferably. When there is nothing and it exceeds ten-mol %, a molecular weight fall becomes almost practical [ the effectiveness to improvement in distributed stability / become remarkable and ] less less than [ 0.1 mol % ].

[0017] In this invention, into a giant molecule, the polyelectrolyte which makes it exist underwater at the time of manufacture of an acrylamide (meta) system polymer has carboxylate and/or a sulfonate, and means what produces phase separation by the interaction with the acrylamide (meta) system polymer to generate. As such a polyelectrolyte, it is independent in a kind of the anionic partial saturation monomer of said instantiation, or the salt of the polymer obtained by carrying out the polymerization of the two or more sorts is raised, for example. Especially as a polyelectrolyte of this invention, the salt of the acrylic-acid polymer which used the acrylic acid as an anionic partial saturation monomer is desirable.

[0018] Moreover, although especially the class of salt of a polymer is not limited, in this invention, alkali-metal salts, such as sodium salt or potassium salt, are desirable. Therefore, especially as a polyelectrolyte of this invention, the alkali-metal salt of an acrylic-acid polymer is desirable. the neutralization index of the carboxylic acid of a polyelectrolyte, and/or a sulfonic acid -- usually -- 50-100-mol % -- it is 70-100-mol % preferably. Formation of this carboxylate and/or a sulfonate may be performed in the phase of an anionic partial saturation monomer, and you may carry out, after manufacturing a polymer.

[0019] Moreover, if the polyelectrolyte of this invention is within the limits which produces phase separation by the interaction with an acrylamide (meta) system polymer, it will be used together with an anionic partial saturation monomer, copolymerizes at least one sort chosen from amide group content partial saturation monomers, such as a cationic partial saturation monomer of said instantiation, a nonionic partial saturation monomer, and acrylamide (meta), and may be obtained. usually, the total mol sum of the partial saturation monomer from which the rate of the copolymerization monomer used together with an anionic partial saturation monomer constitutes a polyelectrolyte -- receiving -- usually -- less than [ 30 mol % ] -- it is less than [ 10 mol % ] preferably. In exceeding 30-mol %, it is hard to produce phase separation by the interaction with an acrylamide (meta) system polymer.

[0020] Although it cannot generally \*\*\*\*\* since the weight average molecular weight of the polyelectrolyte used for this invention will not be restricted especially if it insolubilizes in water by mixing with an acrylamide (meta) system polymer, but it changes with the presentation of an acrylamide (meta) system polymer, conditions of a polymerization reaction, etc., weight average molecular weight is 100,000 or less, and 5000-50000 are usually desirable. When lower than 5000, phase separation cannot happen easily, and the dispersion liquid of an acrylamide (meta) system polymer are not obtained. When exceeding 100,000, the viscosity of a system becomes moreover,

less practical [ increase and ].

[0021] Next, the manufacture approach of the acrylamide (meta) system polymer of this invention is explained in detail. although the amount of the polyelectrolyte used changes with the presentation of a polyelectrolyte, molecular weight, the presentation ratio of a polymerization nature monomer and polymerization concentration, and other polymerization conditions -- usually -- the weight ratio of a polyelectrolyte and a polymerization nature monomer -- usually -- the former / latter = 10 / 90 - 90/10 -- it is 20 / 80 - 50/50 preferably. If the rate of a polyelectrolyte separates from this range, separation of the acrylamide system polymer obtained by carrying out a polymerization (meta) will not take place, and dispersion liquid will not be obtained.

[0022] Moreover, the water-solution concentration of a polymerization nature monomer is usually 15 - 40 % of the weight preferably five to 50% of the weight. The dispersibility of the dispersion liquid which will be obtained if water-solution concentration exceeds 50 % of the weight, and a fluidity become less practical [ fall and ]. Moreover, at less than 5 % of the weight, the phase separation by the interaction of a polyelectrolyte and an acrylamide (meta) system polymer stops arising, and dispersion liquid are not obtained.

[0023] Except for making a polyelectrolyte exist, various well-known approaches and the same approach can be underwater used for the method of carrying out the polymerization of said polymerization nature monomer, and obtaining an acrylamide (meta) system polymer conventionally. For example, a polymerization nature monomer, a polyelectrolyte, and water are taught to a predetermined reaction container, the usual radical polymerization initiators, such as a redox system polymerization initiator of the form which combined persulfate, such as potassium persulfate and ammonium persulfate, or these and the reducing agent like a sodium hydrogensulfite, can be added, and the dispersion liquid of the acrylamide (meta) system polymer of this invention can be obtained by the approach of warming under churning.

[0024] The amount of the radical polymerization initiator used is set up in consideration of the amount of the water-soluble allyl compound (meta) used etc. in the range which can respond to the molecular weight of the acrylamide system polymer obtained (meta). Usually, the amount of the radical polymerization initiator used is usually 0.01-10-mol % to a polymerization nature monomer. moreover, pH of the polyelectrolyte water solution containing the polymerization nature monomer at the time of a polymerization -- usually -- 6-10 -- it is 7-8 preferably, and it is necessary to set up the neutralization index of the carboxylic acid of said polyelectrolyte, and/or a sulfonic acid so that it may go into this range Since it produces hydrolysis of an acrylamide system polymer in phase separation's stopping being able to happen easily when pH is lower than 6, and exceeding 10 (meta), it is not desirable. Although the temperature of a polymerization changes with presentations and those concentration of the class of radical polymerization initiator, a polymerization nature monomer, and a polyelectrolyte and it cannot generally \*\*\*\*\*, it is usually within the limits of 25-90 degrees C, and a polymerization is performed to the bottom of such a condition for 1 to 12 hours. In addition, on the occasion of a polymerization, chain transfer agents other than a water-soluble allyl compound (meta), a surfactant, a salt, etc. can be used for arbitration as an additive if needed. Although the amount of the additive used changes with applications of the acrylamide system polymer obtained (meta) etc. and cannot be limited especially, generally it is 10 or less % of the weight to a polymerization nature monomer.

[0025] The weight average molecular weight of the acrylamide (meta) system polymer obtained in this way is usually 100,000 to about 20 million. Moreover, an acrylamide (meta) system polymer produces phase separation by the interaction with a polyelectrolyte, and is obtained as dispersion liquid of both mixed constituent particle. The viscosity of the dispersion liquid of the acrylamide system polymer containing this polyelectrolyte (meta) is usually about (25 degrees C) 100-10000cps in 20% of concentration. The distributed object of the acrylamide system polymer containing such a polyelectrolyte (meta) is a gel object which usually contains 30 - 70% of the weight of water. These have dissociated with water and are rich in dispersibility and a fluidity. Although the weight ratio of the polyelectrolyte in a gel object and an acrylamide (meta) system polymer changes with conditions of the preparation ratio of each component, or a polymerization,

they are usually the former / latter  $\approx 50 / 50 - 10/90$ . In addition, although surfacing of a distributed object and separation may produce the obtained distributed object if it carries out long duration gentle placement, as for the distributed object using a water-soluble allyl compound (meta), it is possible as a polymerization nature monomer to make churning re-distribute easily.

[0026] Moreover, the water gel particle of the acrylamide system macromolecule containing a polyelectrolyte (meta) is separable from dispersion liquid with water easily by centrifugal separation, filtration, or other suitable separation approaches. Moreover, water and the separated water gel particle have little moisture content, it is more nearly granular than the water gel polymerization object obtained by the usual aqueous polymerization, and since surface area also becomes large, it is easy to dry. Generally, warm air (for example, 30-90 degrees C) can perform desiccation with the fluid bed etc.

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OPERATION

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[Function] The aqueous dispersion liquid of the high-concentration acrylamide (meta) system polymer which is rich in dispersibility and a fluidity with this invention can be obtained. Although the detail of the operation cannot fully be solved, since the insolubilization phenomenon by the interaction of the acrylamide (meta) system polymer and polyelectrolyte in a water solution is applied to formation of dispersion liquid, it is hard to be influenced of a presentation by the polymer monomer, and distributed stability is good and it is the description that the aqueous dispersion liquid of the hypoviscosity which is rich in a fluidity can be obtained. Moreover, an acrylamide (meta) system polymer is easily separable as a water particle containing a polyelectrolyte. A water particle is not only easy handling, but the solubility over water is good and it can raise as a description that dilution time amount can be shortened. In addition, when a cross-linking partial saturation monomer is used as a polymerization nature monomer, the bridge formation particle dispersion liquid of an acrylamide (meta) system polymer are obtained. Filtration can separate this bridge formation particle easily, and a polyelectrolyte can be removed by washing with water. Furthermore, in manufacture of the dispersion liquid by the interaction of an acrylamide (meta) system polymer and a polyelectrolyte, also when the polymerization nature monomer of the distributed object using a water-soluble allyl compound (meta) is high concentration as a polymerization nature monomer, the condensation between the particles under polymerization can be controlled, and large improvement in distributed stability can be realized.

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[Translation done.]

## \* NOTICES \*

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- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

## EXAMPLE

[Example] Although an example explains this invention concretely below, this invention is not limited to this. In addition, % in each example is weight criteria, and the molecular weight of an acrylamide (meta) system polymer is the value which measured using drainage system gel PAMINEESHON chroma log RAFI, and was converted into polyethylene-glycol molecular weight.

[0029] Example 1 acrylamide 100g, 576.9g (pH8.0, weight average molecular weight 19000 [ about ]) of 26-% of the weight sodium polyacrylate water solutions, and 92.3g (12.7 % of the weight of concentration of a polymerization nature monomer) of ion exchange water A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, 10g of ammonium persulfate water solutions and 10g of 1-% of the weight sodium-hydrogensulfite water solutions are added 1% of the weight through nitrogen gas, and a polymerization is carried out at 40 degrees C under churning for 10 hours. The polymer distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 5500cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 8500000.

[0030] Example 2 acrylamide 170g, 7.2g of acrylic acids, 3.2g of sodium hydroxides, 395.3g (pH8.3, weight average molecular weight 15000 [ about ]) of sodium polyacrylate water solutions, and 274.3g (20.6 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, isopropyl alcohol 7.5g and 14.2g of 10-% of the weight ammonium persulfate water solutions, and 5.7g of 10-% of the weight sodium-sulfite water solutions are added through nitrogen gas, and a polymerization is carried out at 40 degrees C under churning for 4 hours. The polymer underwater distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 1200cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 5400000.

[0031] Example 3 acrylamide 100g, 37.5g of the fourth class ghost water solutions of 81-% of the weight dimethylaminoethyl methacrylate methyl chloride, 232.6g (pH8.3, weight average molecular weight 15000 [ about ]) of sodium polyacrylate water solutions, and 281.3g (19.9 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, isopropyl alcohol 4.7g and 0.38g of 10-% of the weight ammonium persulfate water solutions, and 0.14g of 10-% of the weight sodium-sulfite water solutions are added through nitrogen gas, and a polymerization is carried out at 80 degrees C under churning for 4 hours. The polymer distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 200cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 12 million.

[0032] Example 4 acrylamide 200g, 8.5g of acrylic acids, 3.8g of sodium hydroxides, 199.3g (pH8.3, weight average molecular weight 15000 [ about ]) of sodium polyacrylate water solutions, and 231.6g (30.5 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached

nitrogen installation tubing, the thermometer, and the reflux condenser. Keeping at 35 degrees C, except for the dissolved oxygen in liquid, isopropyl alcohol 9g and 16.7g of 10-% of the weight ammonium persulfate water solutions, and 6.8g of 10-% of the weight sodium-sulfite water solutions are added through nitrogen gas, and a polymerization is carried out at 80 degrees C under churning for 4 hours. The polymer distributed by the shape of a particle was obtained. The viscosity of these dispersion liquid was 6800cp at 25 degrees C. Moreover, the weight average molecular weight of the generated acrylamide system polymer was 2100000.

[0033] Example 5 acrylamide 100g, isopropyl alcohol 3.3g (3.9mol for acrylamide %), 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 164.1g (19.6 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer was obtained by adding 8.0g of ammonium persulfate water solutions, and 3.3g of 10-% of the weight sodium-pyrosulfite water solutions 10% of the weight except for the dissolved oxygen in liquid through nitrogen gas, and carrying out a polymerization at 45 degrees C under churning for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 3800cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 2 million.

[0034] The particle which consists of an acrylamide system polymer and sodium polyacrylate was separated from dispersion liquid by carrying out filtration under reduced pressure (100 or more mmHg) of the acrylamide system polymer dispersion liquid compounded in the example 6 example 2 using a glass filter. The particle was the water gel which contained 43% of the weight of water with the mixture of an acrylamide system polymer (85 % of the weight) and sodium polyacrylate (15 % of the weight). The obtained granular water gel could be ground easily and has been dried in about 30 minutes 65-degree-C time amount with the fluid bed dryer.

[0035] While putting 100g of sodium acetate, and 200g of ion exchange water into the reaction container used in the example of comparison 1 example 1 as acrylamide 100g and a low-molecular electrolyte and keeping at 35 degrees C When 10g of ammonium persulfate water solutions and 10g of 1-% of the weight sodium-hydrogensulfite water solutions were added 1% of the weight except for dissolved oxygen through nitrogen gas and the polymerization was performed at 50 degrees C under churning, the viscosity of reaction mixture became extremely high, and even if it carried out the polymerization for 10 hours, dispersion liquid were not obtained with the gel lump.

[0036] In the reaction container used in the example of comparison 2 example 1, acrylamide 75g, 42.5g of ammonium sulfates, 42.5g of diammonium hydrogenphosphate, 7.5g (weight average molecular weight 15000 [ about ]) of sodium polyacrylate, and 200g of ion exchange water are put in. If 0.75g of ammonium persulfate water solutions and 0.7g of 1-% of the weight sodium-hydrogensulfite water solutions are added 1% of the weight except for dissolved oxygen through nitrogen gas and a polymerization is performed at 40 degrees C under churning, keeping at 35 degrees C, the viscosity of reaction mixture is with a gel lump, even if it becomes extremely high and carries out a polymerization for 10 hours. Dispersion liquid were not obtained.

[0037] Example 7 acrylamide 100g, allyl alcohol 3.2g (3.9mol for acrylamide %), 232.6g (pH8.0, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 164.3g (20.2 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer distributed by the shape of a particle was obtained by adding 3.3g of pyrosulfurous acid hydrogen sodium water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 680cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 1090000.

[0038] Example 8 acrylamide 100g, 7.9g (3.9mol for acrylamide %) of sodium allylsulfonate, 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions,

and 159.5g (21.1 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer underwater distributed by the shape of a particle was obtained by adding 3.3g of sodium-pyrosulfite water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 1450cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 1740000.

[0039] Example 9 acrylamide 100g, 1.1g (0.5mol for acrylamide %) of meta-sodium allylsulfonate, 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 166.3g (19.8 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer distributed by the shape of a particle was obtained by adding 3.3g of sodium-pyrosulfite water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 460cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 1190000.

[0040] Example 10 acrylamide 100g, 8.6g (3.9mol for acrylamide %) of meta-sodium allylsulfonate, 232.6g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 58.9g (26.4 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The polymer distributed by the shape of a particle was obtained by adding 3.3g of sodium-pyrosulfite water solutions except for the dissolved oxygen in liquid 8.0g of 10-% of the weight ammonium persulfate water solutions, and 10% of the weight, and carrying out a polymerization at 45 degrees C under churning through nitrogen gas, for 3 hours, keeping at 20 degrees C. The viscosity of these dispersion liquid was 5900cp at 25 degrees C. The weight average molecular weight of the generated acrylamide system polymer was 120000.

[0041] Example 11 acrylamide 100g, methylenebis acrylamide 1.1g (0.5mol for acrylamide %), 8.6g (3.9mol for acrylamide %) of meta-sodium allylsulfonate, 464.5g (pH8, weight average molecular weight 19000 [ about ]) of sodium polyacrylate water solutions, and 424.7g (10.9 % of the weight of concentration of a polymerization nature monomer) of ion exchange water 43% of the weight A stirrer, It puts into 1L reaction container which attached nitrogen installation tubing, the thermometer, and the reflux condenser. The water dispersion of an acrylamide cross-linked-polymer particle was obtained by adding 8.0g of ammonium persulfate water solutions, and 3.3g of 10-% of the weight sodium-sulfite water solutions 10% of the weight except for the dissolved oxygen in liquid through nitrogen gas, and carrying out a polymerization at 45 degrees C under churning for 3 hours, keeping at 20 degrees C. Furthermore, these dispersion liquid were carried out the \*\* exception, and the true spherical particle of a bridge formation acrylamide system polymer with a particle diameter of about 100 microns was obtained by drying among washing and the type dryer of the \*\* style with a lot of water in 105 degrees C and 2 hours.

[0042] The situation under polymerization in an example 5 and examples 7-11, the situation after the standing of the dispersion liquid of the obtained acrylamide system polymer, the viscosity after re-distribution, and redispersible evaluation are shown in Table 1.

[0043] : (Situation under polymerization) At the time of polymerization termination, condensation of the distributed object in the inside of dispersion liquid, and a \*\*\*\* wing and a reaction container wall surface and adhesion were observed by viewing, and were evaluated.

[0044] : (Situation after standing) The obtained dispersion liquid were put at 25 degrees C after polymerization termination for 48 hours, and condensation of the distributed object in the inside of dispersion liquid and a container wall surface and adhesion were observed by viewing, and were evaluated.



[0045] : (Viscosity after re-distribution) After putting dispersion liquid at 25 degrees C for 48 hours, the whole was agitated with the stirrer for 5 minutes, and the viscosity of dispersion liquid was measured by the Brookfield viscometer. In addition, all over Table 1, the result of having measured the viscosity of the obtained dispersion liquid by the Brookfield viscometer after polymerization termination is shown as (viscosity in front of standing).

[0046] : (Redispersible evaluation) Viewing observed and estimated the situation after re-distribution.

[0047]

[Table 1]

|       | 重合中の状況 | 静置後の状況 | 静置前の<br>粘度<br>(c p) | 再分散後<br>の粘度<br>(c p) | 再分散性<br>の評価 |
|-------|--------|--------|---------------------|----------------------|-------------|
| 実施例 5 | 凝集物付着  | 分離、凝集  | 3 8 0 0             | 2 0 5 0              | 不良          |
| 実施例 7 | 付着物なし  | 分離     | 6 2 0               | 5 5 0                | 良好          |
| 実施例 8 | 付着物なし  | 分離     | 1 4 5 0             | 1 4 0 0              | 良好          |
| 実施例 9 | 付着物なし  | 分離     | 4 6 0               | 4 7 5                | 良好          |
| 実施例10 | 付着物なし  | 変化なし   | 5 9 0 0             | 6 0 0 0              | 良好          |
| 実施例11 | 付着物なし  | 分離     | 1 5 8               | 1 5 0                | 良好          |

[Translation done.]